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LOW ENERGY SPUTTERING

Final Report

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Charles B. Cooper
Physics Department
University of Delaware
Newark, Delaware 19711

February, 1968

U. S. Army
Ballistic Research Laboratories
Aberdeen Proving Ground, Maryland 21005

APG Contract No. DA-18-001-AMC-748 (X)

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ABSTRACT

Various experiments on low energy sputtering (the ejection of particles from a solid surface under ion bombardment in a vacuum) have been performed. Apparatus has been constructed to coat the tip of Ballistic Research Laboratory thermocouples with a 2 micron layer of nickel obtained by sputtering, for evaluation and comparison with similiar films formed at BRL by other means. Several thermocouples were coated. Several compound semiconductor crystals were sputtered. This work included measurements of sputtering yields, mass spectrometric study of the sputtered particles, and a study of the angular distribution of the sputtered atoms. The angular distribution of sputtered atoms from metallic single crystals was studied, as a function of target temperature, of bombarding ion energy, of the angle of incidence of the bombarding ion, and finally at very low ion energies. Instrumentation work was done on the measurement of the average kinetic energy of particles sputtered from metallic targets. Mass spectrometric measurements of the sputtering of a Cu single crystal were made, and a search for negative sputtered ions carried out. The sputtering yield of single crystal faces of Ag at low ion energies were measured. Finally, preliminary work was carried out on the low energy sputtering of insulators.

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A. BRL Thermocouple Sputtering.

The work reviewed in this report is described in detail in a Thesis[1] written by Mr. Stanley T. Ockers. A copy of this thesis has been sent to BRL.

(1) Introduction

The purpose of this work was to coat the tip of BRL thermocouples with a two micron layer of nickel by sputtering. The purpose of the present work was to determine if a sputtered film would adhere more tenaciously to the thermocouple during use, than the evaporated films being prepared at BRL.

(2) General Description of Sputtering Method

A schematic drawing to illustrate the method of sputtering is given in Figure 1.

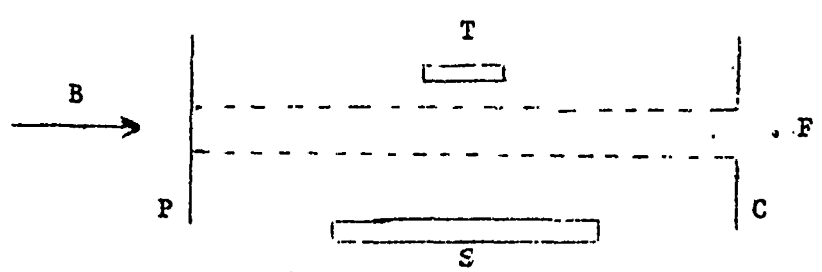

Figure 1

The apparatus is placed in a bell jar vacuum system capable of being evacuated to a pressure of about 10^{-6} torr. Electrons are emitted from a heated tungsten filament F, and drawn by a positive voltage through a slit C into the dotted region shown. The electrons, which were collimated by the magnetic field B, strike the plate P and are collected there. After evacuation, argon gas is admitted into the system to a pressure of about 10^{-3} torr. An argon arc is then struck by the electron beam. An argon plasma is formed in the region between C, S, P, and T. T is a target of material to be sputtered, nickel in this case. It is placed as shown, like a large Langmuir probe, in the argon plasma. When it is biased negatively with respect to the plasma, it is bombarded with argon ions, and sputtered. The sputtered nickel atoms cross the plasma, and form a condensed film on the substrate S. S would be the BRL thermocouple tip, if the run were being made to coat the latter. Refinements on this method were made as described below.

(3) Preliminary Work

Preliminary runs were made using a nickel target to test how well nickel would sputter. The sputtering yield (number of sputtered atoms/number of incident ions) of nickel is moderate, being about 1.5 at an argon/ion energy of 500 eV. Substrates of Fe, Al_2O_3, Al foil, and glass were used. A nickel film could be sputtered on each. However, the magnetic nickel target affected the arc plasma, and the sputtering yield was low. It appeared that considerable time would be required to lay down a two micron layer as required. Work as follows was done to improve the sputtering source.

(4) Instrumentation

I. The Vacuum System

The vacuum system used was a CVC bell jar system, with an 18" bell jar, evacuated by a 6" oil diffusion pump, with a liquid nitrogen trap, backed by a mechanical pump. DC-705 silicon oil was used in the diffusion pump. The pressure was measured with an Alpert type ionization gauge, and with a thermocouple gauge on the forepump line. Argon was bled into the system through a Granville-Phillips type C valve.

II. Heater

A heater was required to hold the BRL thermocouples. After some experimentation it was found that a BN heater, wound with tungsten heating wire was satisfactory. BN has good vacuum properties, a high electrical insulation, and a rather good heat conductivity. The heater was in the form of a cylinder, with a tapped hole in the center in which the thermocouple could be screwed.

III. Sputtering Source

Much experimentation was carried out to increase the current in the ion beam from the sputtering source, so as to maximize the rate of sputtering. Enlarged slits (C, Figure 1) were tried, with and without a W mesh grid arrangement; thoriated filaments were used in place of the tungsten filaments. No improvement resulted. Two filaments, one at C, and one at P (Figure 1) were used, and this increased the arc density materially. Parabolic carbon reflectors were placed behind the filaments to increase the flow of electrons into the arc region. Various insulators around the target were used, and BN found to be the most satisfactory. It also became apparent that very good alignment of slits and filaments were required.

The magnetic property of the nickel target caused trouble, since it tended to bow the magnetic field, and distort the arc plasma. It was discovered that by using a very thin nickel target, the magnetic effect could be reduced for minimum interference with the arc. Additionally, it was found that a rather weak magnetic field (B, Figure 1) was required because of the magnetic property of the iron BRL thermocouple.

The final sputtering source decided on, and built, after the above experimentation is shown in Figure 2.

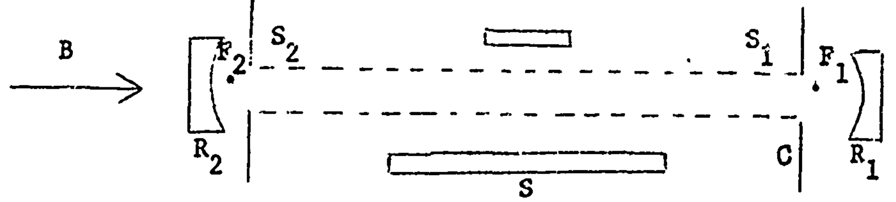

Figure 2

This source is similar to that in Figure 1, except it has two filaments, F_1 and F_2, two carbon electron reflectors R_1 and R_2, and two slits, S_1 and S_2. The performance of this source is tabulated below.

In order to increase the plasma density, and reduce the sputtering time, a "high intensity" sputtering source was built. This is not described in detail here, since it was found not to be practical without much further development. If is described in the Third Quarterly Report, and illustrated in that report in Figure 2, page 3. In essence it is a bigger unit, using a larger filament, heated with 50 amperes of current, and with an arc drawing a large current in the region of 3 to 6 amps. However this unit, due to the power involved, ran hot, and would require extensive water cooling. Also, it was found to be "dirty", and the nickel films formed, although thick, were highly contaminated.

IV. Power Supplies

Power supplies were required for heating the filaments, for the filament to slit voltages, for the target voltage. These were originally regulated supplies. However, it was discovered that the target voltage could be run at a higher figure, without arcing, by using an unregulated voltage supply. Presumably, in the latter case, when an arc started, the supply voltage dropped, and the arc was extinguished.

(5) Film thickness Measurements

A two micron film of nickel on the BRL thermocouple tips was required. It was therefore necessary to know the thickness of film deposited under various sputtering conditions.

To determine the average film thickness, nickel was sputtered onto thin Al foils, which were weighed before and after. The film thickness could thus be determined. The weighing was done on a sensitive chemical balance, and also by using a sensitive quartz spring.

Since the sputtering target is almost a point, the film will not be of uniform thickness over an area equal to that of the area of the tip of the BRL thermocouples. To investigate this non-uniformity a film was deposited on a glass substrate. The film on the glass was then investigated using an optical transmission densitometer, with which the

film thickness as a function of distance across the film, could be measured. It was discovered that (over an area the size of the BRL thermocouple tip) the film thickness at the center was about 1.07 that of the average film thickness.

To investiagte the linearity of film thickness with time of sputtering, runs were made at various sputtering times. It was found that the film thickness was about linear with time over a sputtering time interval of one-half to about four hours.

Film thicknesses were measured with various arc conditions, such as filament emission current, electron accelerating voltage, target voltage, and target current. The purpose here was to be able to estimate film thickness, under a given sputtering condition, without having to weigh the substrate.

Using the above information, it is possible to estimate the thickness of nickel film on the BRL thermocouple tips, if arc conditions, time of run, etc. are noted. Typical figures are tabulated below.

(6) <u>Final Assembly, and Sputtering Conditions</u>

The final design of the sputtering source is shown in Figure 2. It appears that this design gives the maximum sputtering output of this general type of source. Using this source, the following conditions of operation were measured:

	Condition 1	Condition 2
V (Filament to slit)	150 volts	150 volts
V (Target to plasma)	1500 volts	1100 volts
I (Filament to slit)	180 ma	180 ma
I (Target)	4.2 ma	5.2 ma
Argon pressure	1 micron	1 micron
Target-substrate distance	1 cm	1 cm
Target-plasma distance	2 mm	2 mm
Target: Polycrystalline nickel	3/16" diam.	3/16" diam.
Rate of deposition of sputtered film onto substrate	60,000 Å/hr.	35,000 Å/hr.

It was found that Condition (1) above was somewhat unstable in operation due to arcing; Condition (2) however led to stable operation without arcing.

Thus, under the above conditions, a nickel film 2 microns thick, could be deposited on the BRL thermocouple tip in a sputtering time of about 20 to 35 minutes.

(7) Sputtering of BRL Thermocouple Tips With Nickel Film

Using the apparatus shown in Figure 2, and approximate operating conditions (2), of paragraph (6), three BRL thermocouple tips were coated with a nickel film by sputtering.

In each case the surface of the thermocouple tip was pre-cleaned by sputtering it with argon ions. It was biased negatively (about 200 volts), so that its surface was bombarded with argon ions from the arc. This was carried out for periods of 1 to 3 minutes. It was thought that this pre-sputtering would clean and roughen the thermocouple tip surfaces.

Voltage was then removed from the thermocouple, and applied to the nickel target. The thermocouple then acted as the substrate, and a nickel film sputtered from the target was deposited on it. Thermocouples with films of 7, 4, and 2 micron thicknesses were prepared. They have been delivered to Aberdeen for evaluation.

First two thermocouples coated were not in good condition, having been through several runs at BRL. Their resistance was very low, and the insulation in poor shape. Although of doubtful further use as thermocouple, they were sputtered for an evaluation of the sputtered film. The third thermocouple was in better shape before sputtering although it too had been previously used.

(8) Summary

A low pressure, thermonically maintained, magnetically contained argon arc plasma sputtering source has been developed for sputtering of nickel. When run under stable conditions, it is capable of coating a substrate with a 2 micron film of nickel in about 30-40 minutes sputtering time. Three BRL thermocouple tips have been coated with a sputtered nickel film using this apparatus and delivered to BRL for evaluation. For a good comparison of the sputtered film as compared with a film prepared by another method, such as evaporation, probably new, clean, smooth thermocouples should be used.

B. Sputtering of Compound Semiconductors

(I) Sputtering Yields of Several Compound Semiconductors

This work is described in a publication by J. Comas and C. B. Cooper[2].

(A) Introduction

Sputtering yields of several single crystal compound semiconductors were measured, under argon ion bombardment, with energies ranging from 75 to 600 eV. The semiconductors were: CdS($10\bar{1}0$); GaAs ($\bar{1}\bar{1}\bar{0}$); SiC (0001); GaP (111); PbTe (111); and InSb (orientation unknown).

(B) Apparatus

The apparatus for sputtering was similar to that diagrammed in Figure 1. The additional feature for this work was that the target

was suspended from a sensitive quartz spring (sensitivity, 1.33 mm/mg). The weight loss of the target due to sputtering could then be measured using this spring, in situ. From the weight loss, and the bombarding ion current, the sputtering yield could be calculated.

(C) Procedure

The vacuum system was pumped down to base pressure; argon gas was admitted to a pressure of about 3×10^{-3} torr so that an argon arc could be struck. The targets were biased negatively, bombarded with argon ions, and thus sputtered. Sputtering times ranging from one to ten hours were required. The targets were weighed before and after sputtering by the quartz spring balance. The procedure was then repeated for a different ion energy. Separate experiments using a Langmuir probe indicated that the plasma potential was about 7 volts positive. A thermocouple attached to the target indicated that the target temperature did not exceed 100°C.

(D) Results and Discussion

Yields at 500 eV ion energy ranged from 0.40 for SiC to 1.30 for PbTe. Graphs showing yields for all the compounds measured, vs. ion energy are given in reference 2.

(II) Mass Spectrometric Study of Sputtering of Single Crystals of GaAs by Low Energy Argon Ions.

(A) Introduction

In a continuation of the sputtering of compound semiconductors, a mass spectrometric study of the sputtering of single crystals of GaAs was made, to determine the e/m of the ejected particles, to see whether the ejection was atomic or molecular, and if atomic whether it was stiochiometric. This work is described in a publication by J. Comas and C. B. Cooper[3].

(B) Apparatus and Procedure

A mass spectrometer specially constructed for a study of neutral sputtered particles was used in the work. This instrument is described in a publication by J. R. Woodyard and C. B. Cooper[4].

(C) Results and Discussion

Single crystals of GaAs ((110), (111), and $(\bar{1}\bar{1}\bar{1})$) were sputtered by normally incident argon ions (energy from 0 - 140 eV) in the source of the mass spectrometer. For each face, approximately 99.4% of the collected ions were neutral Ga and As atomic species; the balance were neutral GaAs molecules. No neutral Ga_2, As_2, or $(GaAs)_2$ molecules, nor negative Ga^-, As^-, or $(GaAs)^-$ ions, with the characteristics of sputtered particles were detected. Sputtering "yields" for the three faces were found to be: $(111) \approx (\bar{1}\bar{1}\bar{1}) > (110)$. Full details of the experiment can be found in reference 3.

(III) Angular Distribution of Argon Sputtered Particles from the (111), $(\bar{1}\bar{1}\bar{1})$, and (110) Faces of GaAs Single Crystals, vs. Temperature and Ion Energy.

(A) Introduction

The (111), $(\bar{1}\bar{1}\bar{1})$, and (110) faces of a GaAs single crystal were sputtered by normally incident argon ions. The angular ejection of the sputtered material was investigated by forming Wehner type "spot patterns".

(B) Apparatus and Procedure

The apparatus used was similar to that shown in Figure 1, consisting of a magnetically confined, thermionically sustained argon arc. The target to be sputtered is immersed in the arc like a probe and biased neagtively. It is sputtered with argon ions from the arc. The sputtered material condensed in the form of a thin film on glass collectors. The temperature of the target can be varied and measured; and the argon ion energy can be varied from 0 to about 600 eV.

(C) Results and Discussion

From the (110) face of the GaAs crystal an intense sputtered spot was found, normal to the crystal surface. This corresponded to ejection from the $\langle 110 \rangle$ direction of the crystal. This spot did not materially change over the full range of target temperature (50°C to 525° C), and over the full range of argon ion energy (100 to 600 eV).

From the (111) face of the GaAs crystal a distinct spot pattern, of three-fold symmetry, was found. The pattern was visible at all energies and temperatures. However, its clarity improved as the temperature of the target was raised. The three spots were due to sputtered particles being emitted along the $\langle 110 \rangle$ directions of the crystal.

From the $(\bar{1}\bar{1}\bar{1})$ face of the GaAs crystal, preferential ejection does not appear, as with the (111) face. The emission is more cosine like. This is true at all ranges of temperature and ion energy studied.

A similar series of runs were made on the (111) and $(\bar{1}\bar{1}\bar{1})$ faces of a GaP crystal. Results similar to those obtained with GaAs were found.

Recent measurements on sputtering of InSb by Anderson[5] and on LEED experiments[6] and on field emission studies[7] indicate that the (111) face of these crystals retains a crystalline structure, but that the $(\bar{1}\bar{1}\bar{1})$ face appears to have a more amorphous like structure. This agrees with our observations of spots from the (111) face, but not from the $(\bar{1}\bar{1}\bar{1})$.

C. Angular Distribution of Sputtered Atoms from Metallic Single Crystals

(I) Angular Distribution of Sputtered Atoms as a Function of Temperature

This project involves the sputtering of a (110) face of a copper single crystal by a low energy argon ions. The purpose of the experiment is to investigate the effect of target (i.e. Cu) temperature on the angular distribution of the sputtered Cu atoms. Copper single crystals show preferential sputtering along directions parallel to the close packed directions in the crystal. Because theory indicated that sputtering (low energy) is a momentum transfer process between atoms, the ejection should be affected by the crystal temperature.

The apparatus used is basically like that shown in Figure 1. In addition, provision is made for a heater to heat the Cu target to the required temperatures, while sputtering is going on. A thermocouple is also included to read the target temperature. The substrate in this case is a glass plate on which the sputtered atoms are condensed.

The Cu target was sputtered at argon ion energies of 50, 100, and 150 eV. At each of these energies, target temperatures of 300°, 600°, and 900° C were used. A total of 20 runs was made.

After each run, the glass substrate with the sputtered film condensed on it was placed in an optical transmission densitometer. This instrument was used to measure the thickness of the Cu film on the glass, as a function of position along the glass. Three directions on the glass were scanned. The results gave a profile of film thickness. From this, and the geometry of the system, information on the angular distribution of the sputtered copper atoms can be obtained.

It was found that the preferential ejection of atoms (approximately parallel to the close packed directions in the crystal) persisted to the highest temperatures used. This is noteworthy, since at 900°C, the Cu crystals are approaching their melting point, and lattice vibrations are rather strong. It remains to make a quantative analysis of the densitometer scans, to attempt to relate the broading of the "spot patterns" to the amplitude of the bulk and surface atom vibrations of the crystal.

(II) Angular Distribution as a Function of Bombarding Ion Energy

It is known that when single metallic crystals are bombarded normally with inert gas ions, the sputtered crystal atoms leave the crystal surface in preferential directions, depending upon the crystal structure and orientation.

A convenient apparatus for this experiment is similar to that diagrammed in Figure 1. The target to be sputtered is a single crystal. The substrate on which sputtered material is gathered is a glass plate. The sputtered atoms thus form a thin sputtered film on the glass.

If the face of the copper single crystal being sputtered is (100), the atoms will be sputtered preferentially in four directions from the crystal; approximately along the four close packed crystal directions. The film formed on the glass substrate thus has the appearance of four spot ("spot patterns") on the corners of a square. The four close packed directions in the crystal make angles of 45° with the surface. It has been found however, that the sputtering spot patterns are not at exactly 45°. The angles of these seem to vary from about 35° to 44°, depending upon the observer. The purpose of this work was therefore to make a systematic study of these angles as a function on bombarding ion energy.

The Cu (100) crystal was put on the G.E. x-ray machine to check its orientation by a Laue pattern. It was determined to be within 5° of the (100) face; and the azimuthal angle of the crystal was marked with a scratch. The crystal was then put in the sputtering source, and bombarded with argon ions. Ion energies of 50, 75, 100, and 125 eV were used. Films were formed by sputtering onto the glass substrates at each energy. These glass plates (with the sputtering thin film "spot patterns" on them) were then analyzed using an optical transmission densitometer. With this, the thickness of the sputtered deposit could be measured, by scanning across the plates, as a function of the distance along the plates. The center of the "spots" could thus be located. By considering the geometry of the sputtering source, the angle of emission of the sputtered atoms from the crystal could be determined.

The estimated angles of ejections were found to be as follows:

Ion Energy (eV)	Estimated Angle
50	40°54'
75	40°34'
100	40°12'
125	38°48'

There thus seems to be evidence: (1) that the angles of ejection is not 45°; and (2) that the angle of ejection decreases as the bombarding ion energy increases. Since the change in angle is small, further data should be taken before definite conclusions can be drawn.

(III) Angular Distribution as a Function of Angle of Incidence of Bombarding Ion

Referring to the discussion above, it is also of interest to study the angle of emission of sputtered particles from single crystals, when the angle of incidence of the bombarding ions is changed. Work was done to attempt to construct as ion source with a directed beam of ions, whose angle of incidence on the crystal surface could be varied. In this work,

it is the low energy region which is of interest. The energy of the ions should range from about 40 to 125 eV or so. It is a difficult problem to make an ion source of these specifications. The ion current must be fairly large, of the order of several ma/cm^2, in order that the sputtered particles can be detected. However with this large current, and low voltages, the space charge of the ion beam causes it to spread rapidly. Thus a focussed ion beam at these energies is difficult to obtain. Some preliminary work using an ion source as shown in Figure 3 was done.

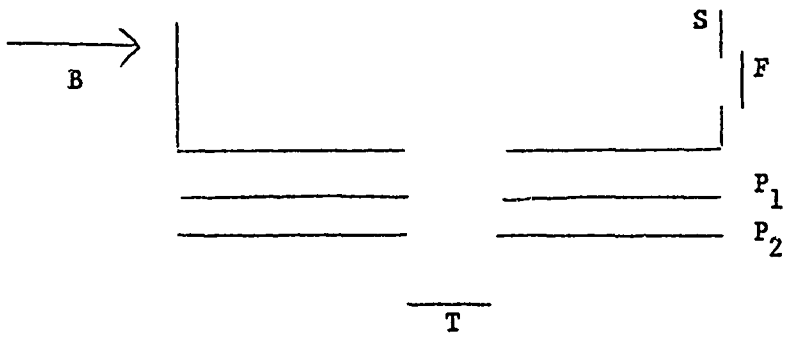

Figure 3

As in Figure 1, a magnetically confined argon arc is struck in the region of the dotted line. Plates P_1 and P_2 are biased so as to draw ions from the arc. Plate P_1 is made negative, and plate P_2 less negative, so that a focussing action will take place. A beam of ions thus goes through the hole in plate P_2. T is a target to monitor this ion beam. As a gas pressure of 10^{-3} torr, and with 75 volts on plates P_1 and P_2, a current to the target of 0.5 ma was measured. Further measurement with a probe in the region of T, to investigate the amount of spread in the ion beam, is required before it can be determined if such an ion source is feasible.

(IV) Search for "Rotation" of Spot Patterns From (100) Faces of f.c.c. Metal Crystals at Very Low Ion Energy

When an f.c.c. crystal (such as Ag (100)) is bombarded normally with noble gas ions, it is well known that the sputtered Cu atoms are ejected preferentially in four directions. These directions correspond roughly to the four close packed directions in the crystal. When the sputtered material is collected on a glass substrate, thus, four "spots" appear.

Some theories of sputtering (those due to Langberg[8] and Schlaug[9], for example) predict that the four "spots" should appear, but that as the energy of the bombarding ions is reduced to a low value, the spot pattern should no longer be along the four close packed directions of the crystal. In addition, Schlaug's theory indicates that there will be a difference in the pattern depending upon whether the bombarding gas ion has a mass less than or greater than the mass of the target atom.

Experiments were run to test these theories. Targets of Cu (100)

and Ag (100) were used; and bombarding gases of Ar and Kr were used. It is noted that the masses of these atoms are in the following order: M(Kr) > M(Cu) > M(Ar); and M(Ag) > M(Kr) > M(Ar).

The sputtering was done with our usual bell jar apparatus. A thermionically sustained, magnetically confined arc was set up using either Ar or Kr. The target to be sputtered was immersed in the arc, and biased negatively. Thus, positive ions fromthe arc bombard it normally, with a high current density, and it is sputtered. The arc voltage was run at about 55 volts for Ar, and about 35 volts for Kr. The target voltage (that is the energy with which the ions hit the target) was about 65 volts in each case. Four experiments were done: Cu(100) was bombarded with Ar and then Kr ions; and Ag(100) was bombarded with Ar and the Kr ions. The sputtered particles were condensed on glass substrates in each case to study the "spot patterns".

In no case was there any evidence of any rotation of the spots. The four usual spots appeared, and they were along directions corresponding approximately to the four close packed directions in the crystal. Thus, we detected no rotation of the spot patterns. This is at variance with the theories mentioned.

(The close packed directions in the crystal were determined by taking Laue x-ray pictures of both the Ag and Cu crystals).

D. Measurements of Velocities of Neutral Sputtered Atoms

Preliminary experiments have shown that the velocity of sputtered atoms is at least an order of magnitude larger than those of evaporated particles. This is evidence in favor of the momentum transfer theory of sputtering, and it is thus of interest to investigate the velocities more closely.

This work, done by undergraduate Willard White, describes development of an apparatus to make these measurements.

Sputtered atoms are mostly neutral, making their detection and measurement difficult. The apparatus used must be capable of detecting a neutral beam.

The sputtering apparatus is similar to that shown in Figure 1. This apparatus is located in a vacuum bell jar with an ultimate vacuum of about 10^{-6} torr. A magnetically confined argon arc is used. Ion drawn from this arc bombard a Cu target and cause it to sputter. The sputtered atoms move toward S.

The particular detection equipment used in this experiment was a sensitive condenser microphone. It was placed at the position of the

"substrate", so that the beam of sputtered particles caused its diaphragm to bend, thus changing the capacity of the microphone.

Due to the fact that the microphone has a much higher sensitivity as an a.c. device at its resonant frequency, a pulsed beam was used. The target voltage used was a square wave of frequency about 7000 c.p.s. Thus the target was bombarded by pulses of ions; and the sputtered beam therefore left the target in pulses. The sputtered atoms which hit the microphone diaphram was thus also pulsed. The diaphragm thus oscillated, with an amplitude proportional to the momentum of the sputtered atoms.

Numerous bugs were found when the experiments were tried, the major one being the noise problem, due to the great sensitivity of the microphone. Therefore good noise insulation was required. Also, it was found that the square wave voltage on the target effected the microphone. To get around this difficulty, two targets were used, one of C and one of Cu. Alternate readings were taken. Since C has an essentially zero sputtering yield the <u>difference</u> between the two signals was taken as being due to the sputtering of the Cu.

An analysis of the system yielded the following equation for calculating the average velocity of the sputtered particles,

$$\bar{v} = \frac{A + (2\,P^2_{r.m.s.} - (\mu d w)^2)^{1/2}}{\mu}$$

In this formula, $\bar{v}$ is the average velocity; A is the amplitude of the electrostatic pressure on the microphone due to the target voltage (measurable); P_{rms} the r.m.s. pressure on the microphone as read by the signal from the microphone amplifier; μ is a factor containing geometrical factors (size of target and diaphragm) and sputtering yields; d is the target to microphone distance; w is the angular frequency of the square wave.

For a copper target bombarded with 100-120 eV argon ions, use of the above formula yielded a value of $11\text{-}12 \times 10^5$ cm/sec., for the average velocity of the sputtered copper atoms.

Measurements made by Wehner[10] using a spectroscopic technique give values of the velocity of the velocity lower than this by a factor of about two.

The conclusion is that this microphone detection system will detect sputtered atoms, and that with further determination of factors such as sticking coefficients, and angular distribution of sputtered atoms, it will yield quantitative data on the average velocity of sputtered atoms.

E. Mass Spectrometric Study of Sputtering of a Cu (110) Single Crystal

Previous work[4] had been done in this laboratory on a mass spectrometric study of the neutral particles sputtered by low energy argon ions from a polycrystalline Cu target. A similar project has now been carried out, using a single crystal Cu target. The crystal used was a Cu (110) crystal. This particular face was chosen since it has been observed in "spot pattern" work that ejection of sputtered particles normal to this surface starts at a higher energy than ejection in other directions. The geometry of the mass spectrometer is such that it measures this perpendicular ejection. A polycrystalline copper sample was also sputtered for checking the instrument against data previously taken.

Sputtered neutral atoms from the Cu (110) crystal were detected, and the yield was measured at argon ion energies varying from about 40 eV, to about 120 eV. These yields were compared with those from the sample. This agrees with observations made on Cu spot patterns. Sputtering yields agreed qualtitatively with those obtained by other observers using different experimental methods.

F. Mass Spectrometric Search for Negative Sputtered Cu Ions

The mass spectrometer built in this laboratory for the study of neutral sputtered particles can also be used for the study of negative particles, both originating in the argon arc of the source, as well as those coming from the target surface. A program is now underway to search for and study any negative particles sputtered from a Cu target by argon ion bombardment. The apparatus used is that previously described[4]. For the present purposes the polarity of the source plates, and direction of field magnet current have been reversed, so that negative ions can be detected.

G. Measurement of Sputtering Yield of Ag (100) and Ag (111) faces at Low Bombarding Ion Energy

Since little data exists on sputtering yields of single metallic crystals at very low ion energies, these measurements were made.

Apparatus similar to that shown in Figure 1 was used. A magnetically confined, thermionically sustained argon arc was set up. The targets to be sputtered were immersed in the arc and biased negatively. They were then bombarded by argon ions from the arc. (100) and (111) faces of Ag single crystals were sputtered. Argon ion energy was varied from about 110 eV down to the lowest energy at which a measureable yield could reasonably be attained -- this was in the neighborhood of 30 eV. The yields were obtained by weighing the targets before and after bombarding, on a chemical balance. The voltage of the filament in the arc was kept low, to avoid having any Ar^{++} ions in the arc. A polycrystalline sample was sputtered as a check on the experimental method.

It was found that the yield of the Ag (100) face was greater than that of the Ag(111) face at all ion energies tested. The yield for the Ag (100) face increased approximately linearly from a value of 0.28 at an argon ion energy of 35 eV, to a value of 1.3 at an ion energy of 110 eV; and the yield of the Ag (111) from a yield of 0.09 at 35 eV to about 0.6 at 110 eV.

H. Sputtering of Insulators

Previous work described in this report has involved sputtering of metals and semiconductors. Preliminary work is now underway to sputter some insulators, such as alkali halides and ZnS. In low energy sputtering, where the bombarding ions are obtained from an arc, as described in this report, the problem with insulator targets is that they rapidly build up a positive charge from the ion bombardment, repelling further ions, and stopping the sputtering. In order to overcome this problem two techniques are being tried as follows.

The general sputtering source is as that diagrammed in Figure 1. A magnetically confined, thermionically sustained argon arc is used. The target to be sputtered is immersed in the arc like a probe. In order to over come the surface charging problem a very thin target was used, and it was heated to about 300°C, so that it would be somewhat electricaly conducting. In preliminary work done so far, this has not resulted in sputtering. However further work is continuing.

In the second method, the voltage applied to the target is r.f. instead of d.c.[11]. Thus in every half cycle, the electrons attracted to the target from the arc neutralize the positive charge built up from the other half of the cycle when the target is being bombarded by ions. An RF voltage source consisting of an amateur radio transmitter has been constructed and tested. It is capable of operating on frequencies of about 7, 14, 21, and 28 megacycles. Preliminary runs have been made with crystals of ZnS and NaCl. In each case there is evidence of sputtering taking place. Work is continuing to improve the efficiency of the source, and investigate the sputtered particles further.

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13. ABSTRACT

Various experiments on low energy sputtering (the ejection of particles from a solid surface under ion bombardment in a vacuum) have ~~been~~ performed. Apparatus ~~has been~~ constructed to coat the tip of Ba~~llistic~~ Research Laboratory thermocouples with a 2 micron layer of nickel obtained by sputtering, for evaluation and comparison with similiar filsm formed at BRL by other means. Several thermocouples were coated. Several compound semiconductor crystals were sputtered. This work included measurements of sputtering yields, mass spectrometric study of the sputtered particles, and a study of the angular distribution of the sputtered atoms. The angular distribution of sputtered atoms from metallic single crystals was studied, as a function of target temperature, of bombarding ion energy, of the angle of incidence of the bombarding ion, and finally at very low ion energies. Instrumentation work was done on the measurement of the average kinetic energy of particles sputtered from metallic targets. Mass spectrometric measurements of the sputtering of a Cu single crystal were made, and a search for negative sputtered ions carried out. The sputtering yield of single crystal faces of Ag at low ion energies were measured. Finally, preliminary work was carried out on the low energy sputtering of insulators. (U)

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